0155.000 ANALYTICAL METHODS HAVING PROVISIONS FOR A ONE-POINT CALIBRATION AND CONTINUING CALIBRATION VERIFICATION CONSTRAINTS POLICY

Level One Arizona Department of Environmental Quality

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PURPOSE

Most analytical methods have established upper and lower control limits for CCV's and when the recovery exceeds those limits the method is considered “out-of-control”. ADEQ is concerned with the assumption that the 'data are not impacted', as reported by laboratories when the upper control limit of a CCV has been exceeded in a non-detect result. Currently, there is no way to differentiate between an instrument that has gained sensitivity and one that has drifted out of control when the upper control limit of a CCV is ignored.

Adherence to this policy will assure that all laboratory-generated data submitted to ADEQ meets regulatory requirements and are legally defensible.

Because ADEQ is a regulatory agency, compliance results must be able to meet all legal requirements. Where CCV requirements are part of the test method and where test methods are part of the regulatory requirements, then the CCV requirements as dictated by the analytical method must be followed.

AUTHORITY

A.A.C. R18-4-106 and R9-14-608.

The EPA methods continue to be written such that upper and lower control limits for the CCV are established and there is no documentation which permits one to ignore the violation of an upper

control limit in light of certain conditions.

DEFINITIONS

Continuing Calibration Verification Standard (CCV)--Consists of an aliquot of reagent water to which known quantities of the method analytes are added by the laboratory. The CCV's purpose is to determine whether the methodology is 'in control' by verifying the linearity of the calibration curve and to assure that the sample results reflect accurate and precise measurements.

Data--For the purposes of this policy, data is defined as raw data (examples include but are not limited to calibration curves, chromatograms, spectras, injection logs, etc.) and does not include laboratory reports. (Contact the QA unit for further information).

POLICY

From a regulator's perspective, a laboratory must follow the method as written to ensure the analytical data generated is defensible and can survive the scrutiny of litigation. ADEQ will not accept test results for regulatory purposes when the CCV's acceptance criteria have been exceeded. This includes sample results where the upper control limit of the CCV has been exceeded and the result is reported as non-detect.

However, in the event a CCV exceeds its control limits for a detect sample, ADEQ allows the laboratory to either 1)recalibrate the entire multi-point curve and reanalyze the samples or 2) perform a one-point calibration as the method permits.

RESPONSIBILITY

The ADEQ QA/QC staff will be responsible, when reviewing data for the purpose of recommending to ADEQ program staff to either accept or reject such data, to ensure that the procedures outlined in this policy are followed.

APPLICABILITY

This policy is only applicable to those methods which provide for a one-point calibration and those water matrices for the analysis of

volatile organic compounds (VOCs), synthetic organic compounds (SOCs), and inorganic compounds (IOCs) analyzed using 40 CFR methods (ex. 200, 500, and 600 series). This policy does not apply to those samples analyzed using SW-846 methods.

LABORATORY PROCEDURES

EPA and the ADEQ QA/QC Unit require that laboratories which elect to recalibrate using a one-point calibration must demonstrate there is adequate instrument sensitivity to detect a peak at the method reporting level for those contaminants. Therefore, to justify reporting sample results as non-detect when the control limits of a CCV have been exceeded, the laboratory must recalibrate using a standard at the method reporting level and re-run all the samples or extracts after that CCV.

The laboratory must detect a significant peak for each analyte reported in the method reporting level standard. A significant peak is considered to be one in which the peak is at least 3 to 5 times the signal to noise ratio (40 CFR, Part 136, Appendix B, Procedure section 1a).

This ADEQ policy provides a means for laboratories to demonstrate that sample results are, in fact, non-detect for target analytes. The method reporting level standard must be analyzed (and determined to be acceptable) before reanalyzing any samples in a run.

Non-detects:

To report a non-detect result using a one-point calibration, the laboratory must meet the following requirement: Establish the absence of a significant peak at the retention time of the target analyte. The absence of a significant peak at the retention time of the target analyte is defined as one whose response is less than that of the analyte present in the low level standard (which must be prepared at the reporting limit) used for the one-point calibration.

Detects:

To report a detect result using a one-point calibration, a laboratory must meet the following requirement: a one-point calibration must be performed so that the concentration of the one-point calibration standard is within ±20% of the concentration of analyte detected in a sample.

ATTACHMENT

STATEMENT OF POSITION

There has been some debate among the laboratory community concerning continuing calibration verification (CCV's) standards and non detect samples. Most analytical methods have established upper and lower control limits for CCV's and when the recovery exceeds those limits the method is considered “out of control”. Recently, there has been a growing consensus among some laboratories that an analytical method is ***not*** out of control if the upper control limit of the CCV is exceeded providing the sample is a non-detect. The reasoning here is that the instrument has somehow "gained" sensitivity and if there were anything in the sample, it would surely have been detected.

The ADEQ QA/QC Unit understands this logic and recognizes that it may true in some cases. However, this is only one of several possibilities. Another possibility is that the analytical method is now out of control. ADEQ is concerned with the assumption that the 'data are not impacted', as reported by laboratories when the upper control limit of a CCV has been exceeded in a non-detect result. Currently, there is no way to differentiate between an instrument that has gained sensitivity and one that has drifted out of control when the upper control limit of a CCV is ignored.

As a regulatory agency, ADEQ cannot assume that each time the upper control limit is exceeded, it is the result of increased instrument sensitivity. Such an assumption can result in the court or the hearing officer invalidating or dismissing the analytical results because an integral portion of the method's quality control has been omitted. The ADEQ Quality Assurance\Quality Control Unit has discussed this subject at length with EPA Region IX's Quality Assurance Management Section. Region IX concurs with the ADEQ's QA\QC Unit’s interpretation. They have further expressed their concern that ignoring established upper control limits for the CCV is not in line with "good laboratory science" and may invite abuse and even laboratory fraud.